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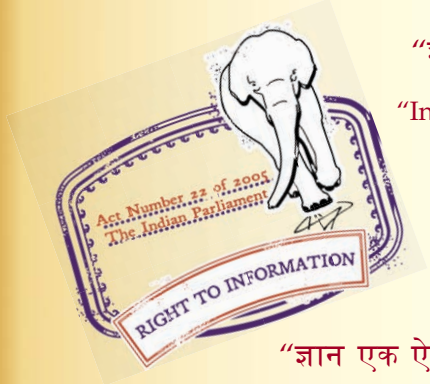

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IS : 7929 - 1975
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METHODS FOR DETERMINATION OF ELECTRICAL RESISTIVITY OF CHEMICAL COKE

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METHODS FOR DETERMINATION OF ELECTRICAL RESISTIVITY OF CHEMICAL COKE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 August 1975, after the draft finalized by the Soild Mineral Fuels Sectional Committee had been approved by the Chemical Division Council.

0.2 ' Chemical coke ' is a general term which is applied to various types of coke other than that used in blast furnaces, cupolas, etc. Chief areas of use of chemical coke are electro-thermal and electro-chemical industries where this coke is used either as a carrier of current or as a source of carbon. Chemical coke is generally used in the form of granules, green moulded shapes or finished carbon blocks.

0.3 In this standard two methods of determination of electrical resistivity have been prescribed. The first is the two-electrode method which is suitable for chemical coke in granular form and the other is the four-probe method which is suitable for green moulded shapes, finished carbon blocks and lumps.

0.4 In the formulation of this standard valuable assistance has been rendered by the Central Fuel Research Institute, Dhanbad, and Geological Survey of India, Calcutta.

0.5 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

---

1. SCOPE

1.1 This standard prescribes methods for determination of electrical resistivity of chemical coke at room temperature.

2. PRINCIPLES

2.1 Two-Electrode Method — This method is applicable to chemical coke in the form of granules of definite size range, compressed to the shape

*Rules for rounding off numerical values (*revised*).

of a cylinder between two metal electrodes in a tube made of a suitable non-conducting material. A direct current is passed through this coke mass and its resistance is measured by a double ohm Kelvin bridge or a potentiometer or a dc vacuum-tube voltmeter. Electrical resistivity (P) is calculated from the following equation:

$$P = \frac{E\,Q}{I\,H} = \frac{R\,Q}{H} \text{ ohm.cm}$$

where

E = potential difference in volts across the electrodes,

Q = cross-sectional area of the coke column in cm^2,

I = current strength in amperes passing through the coke column,

H = height of the coke column in cm, and

R = resistance of the coke column in ohms.

2.2 Four-Probe Method — This method is applicable to chemical coke in the form of green moulded shapes, finished carbon blocks or lumps. This method is independent of the shape or size of the specimen and requires only a linear dimension of about 20 to 200 mm on a flat ground face. By this method electrical resistivity can be measured in a part of the whole bulk of the sample by placing four spring-loaded tungsten or hardened steel probes in a line at equal intervals on the flat ground face of the specimen. A low direct current is passed through the outer probes and the potential difference between the remaining inner two probes is measured by a vacuum tube voltmeter. To minimize the effect of anisotropy, measurements should be made at different points and in different directions on the specimen surface. Electrical resistivity (P) is calculated from the following equation:

$$P = 2\,\pi\,S\,\frac{E}{I} \text{ ohm.cm}$$

where

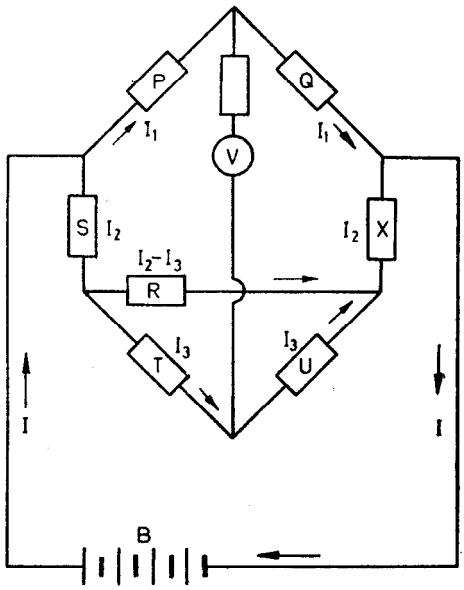
S = distance in cm between the adjacent probes,

E = potential difference in volts between the two inner probes, and

I = current strength in amperes passing through the two outer probes.

3. APPARATUS

3.1 For Two-Electrode Method — The circuit diagram of the set-up is given in Fig. 1. Details of accessories are given in **3.1.1** to **3.1.4.**



I = Total bridge current from the battery

I_1 = Current along 'P' and 'Q' arms

I_2 = Current along 'S' (standard resistance) and 'X' unknown resistance of the sample

I_3 = Current along 'T' and 'U' arms

I_2-I_3 = Current along 'R' (standard low resistance)

X = Unknown resistance (of the sample)

V = Vacuum tube volt meter

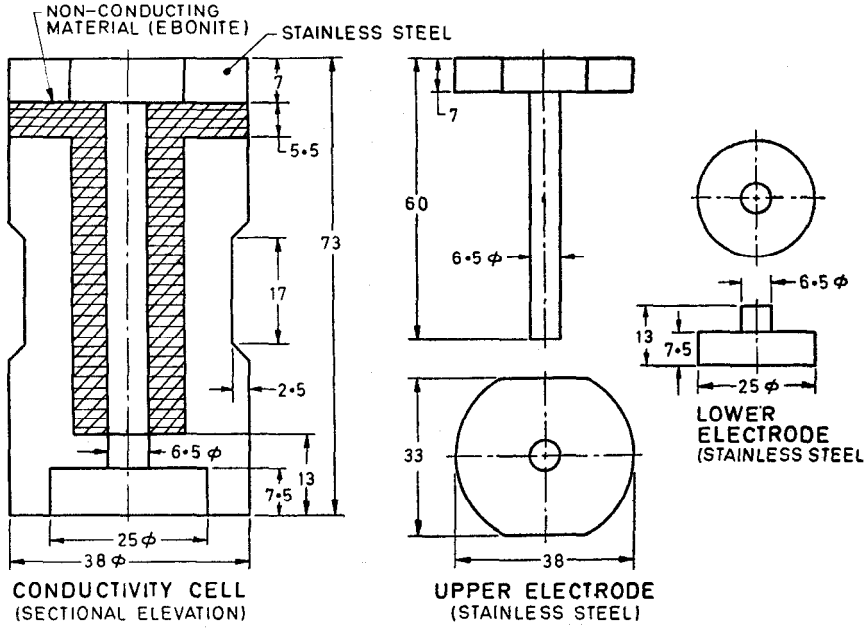
B = Battery (12 volts)

FIG. 1 CIRCUIT DIAGRAM FOR TWO-ELECTRODE METHOD

3.1.1 *Pressure Appliance* — a hydraulic jack provided with a calibrated gauge up to a pressure of 10 000 kN/m² (100 kgf/cm²) with one division equivalent to 100 kN/m² (1 kgf/cm²).

3.1.2 *Specimen Cells* (*or Conductivity Cells*) — made of stainless steel. The inside of the cell is lined with ebonite or steatite or sillimanite. A small cell can hold as small as 0·5 g and a big cell up to 20 g of sample. A stainless steel electrode makes the contact. Details of the small and big conductivity cells are shown in Fig. 2 and 3.

3.1.3 *A Suitable Bridge* — double ohm Kelvin bridge or potentiometer or dc microvoltmeter (vacuum tube microvoltmeter) with necessary accessories for measuring resistance of coke mass with an accuracy of about 0·5 percent. These appliances are suitable for measuring resistance less than 10 ohms. A Wheatstone bridge or a post office box may also be employed.



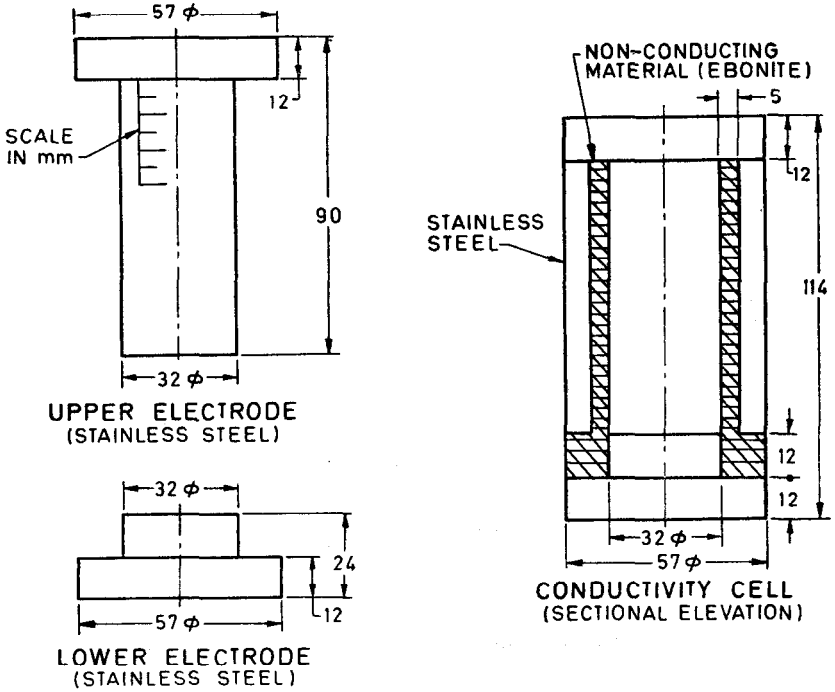
All dimensions in millimetres.

FIG. 2 CONDUCTIVITY CELL (SMALL)

3.1.4 *A Dial Gauge or a Scale* — to measure in millimetres the height of pressed coke column with an accuracy of 0·5 percent.

3.2 For Four-Probe Method — The circuit diagram of the set-up and arrangement of probes are given in Fig. 4 and 5. Details of the apparatus and assembly are given below.

3.2.1 *Probe Instrument* — comprising of the probe head, specimen holder and weight pan (*see* Fig. 6A). The probe head is made of polymerized tetraflouroethylene base material and has four equidistant cylindrical holes in a straight line. These holes lead to rectangular guide slots (*see* Fig. 6B); each slot has a probe holder and a compression coil spring in cylindrical form. The probes, four in number are made of tungsten or steel, have diameter of 0·8 mm and terminate in a sharp tip to minimize area of contact with the specimen. Each probe is brazed on the plate on the probe holder. The probe holders are connected to the terminals. Each probe is attached to its own spring loaded metallic holders. With a view to ensuring proper contact between the probe tips and the secimen surface, the probe holder is weighted by a load of 0·5 to 4 kg placed on top of the vertical shaft of the probe holder. Details of specimen holder are given in Fig. 6C.



All dimensions in millimetres.

FIG. 3 CONDUCTIVITY CELL (BIG)

3.2.2 *Vacuum Tube Microvoltmeter* — *See* **3.1.3.**

3.2.3 *Milliammeter* — of range 0 to 250 mA.

3.2.4 *Travelling Microscope* — to measure distance between the probes (with an accuracy of ±0·01 mm).

3.2.5 *Weights* — 0·5 to 4 kg to be put on the weight pan of the apparatus.

4. TEST SPECIMEN

4.1 For Two-Electrode Method — Carbon or coke particles lying in the size range 0·21 and 0·42 mm are prepared from representative samples [*see* IS : 436 (Part II)-1965*]. Any coke dust adhering to the sized fraction is removed by sieving on a sieve shaker for about 20 minutes. The prepared specimen is then dried in a vacuum air-oven at a temperature

*Method for sampling of coal and coke: Part II Sampling of coke (*revised*).

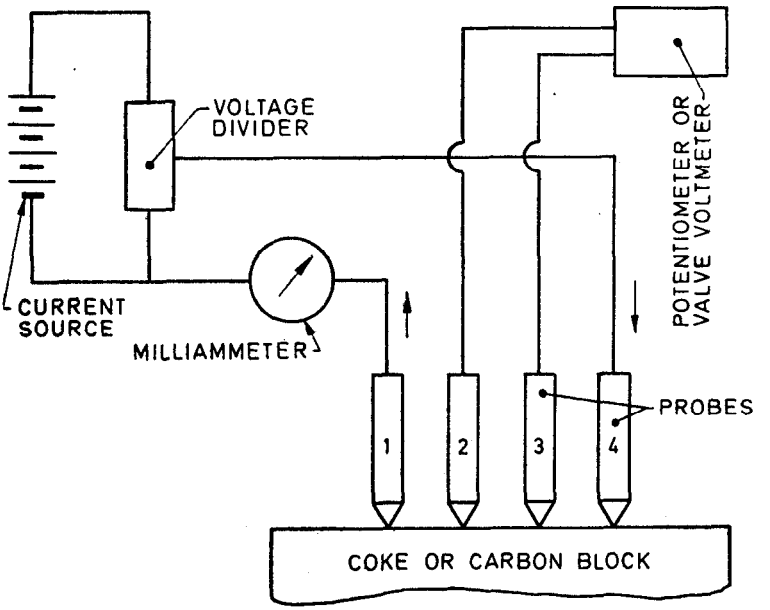
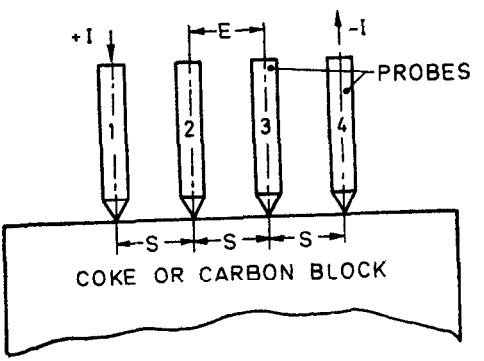


FIG. 4 CIRCUIT DIAGRAM FOR FOUR-PROBE METHOD



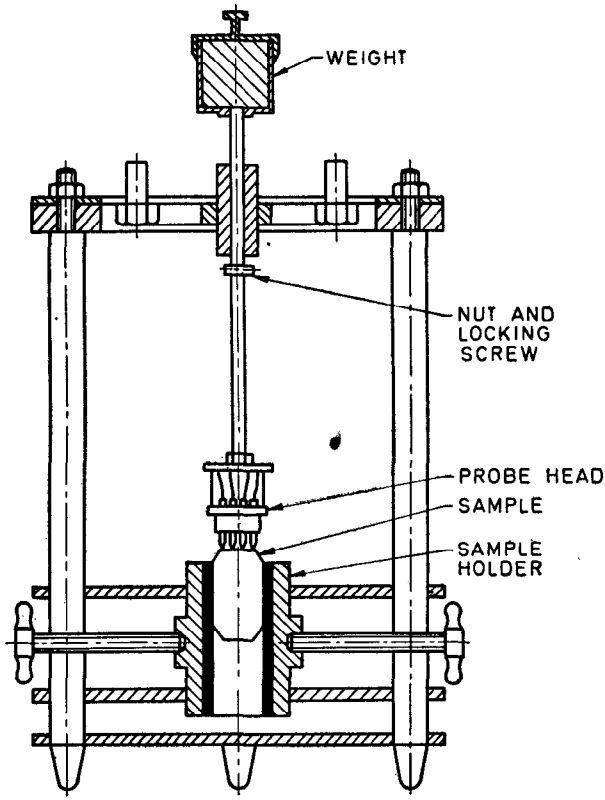
I = Current passing through probes 1 and 4

E = Potential difference between probes 2 and 3

S = Distance between adjacent probes

NOTE — 1, 2, 3 and 4 are probes.

FIG. 5 ARRANGEMENT OF PROBES

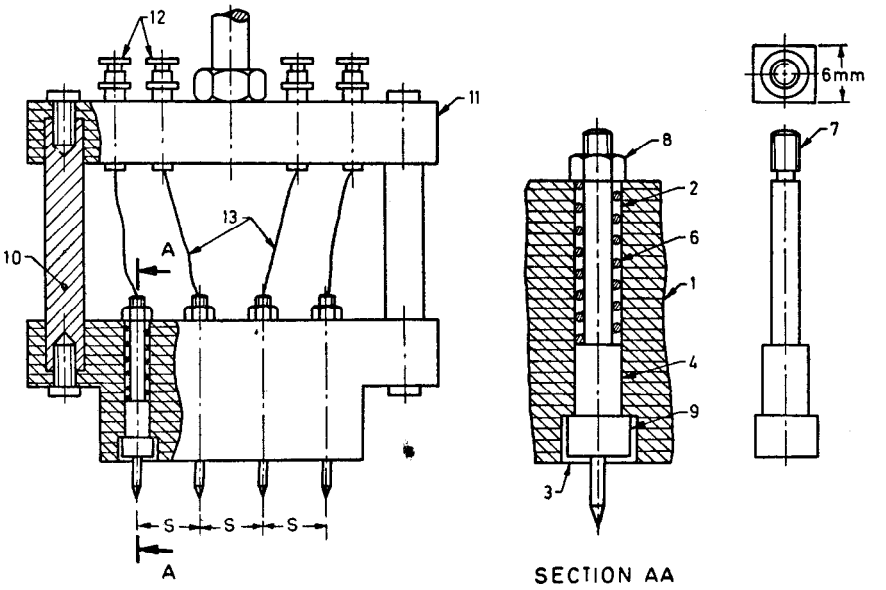


6A Assembly of Four-Probe Instrument

FIG. 6 FOUR-PROBE INSTRUMENT — *Contd*

of 150 to 200°C for a period of 2 hours to remove moisture and then cooled in a desiccator to room temperature and stored in it until required for testing.

4.2 For Four-Probe Method — Specimens of green moulded shapes, finished carbon blocks, coke lumps or blocks having linear dimensions ranging from 20 to 200 mm independent of shape or size (including oval shaped briquettes) are ground on a grinding machine or a glass plate to provide a flat surface to accommodate the four probes. The prepared specimen is then dried in a vacuum air-oven at a temperature of 150 to 200°C and cooled and stored as mentioned in **4.1.**



1. Transparent plastic material
2. Cylindrical holes for the probe
3. Guide slots
4. Probe holder
5. Probe
6. Compression coil spring
7. Cylindrical shank
8. Setting nut
9. Endplate on the probe holder
10. Attaching rod between probe head and terminal plate
11. Terminal plate
12. Terminal
13. Leads

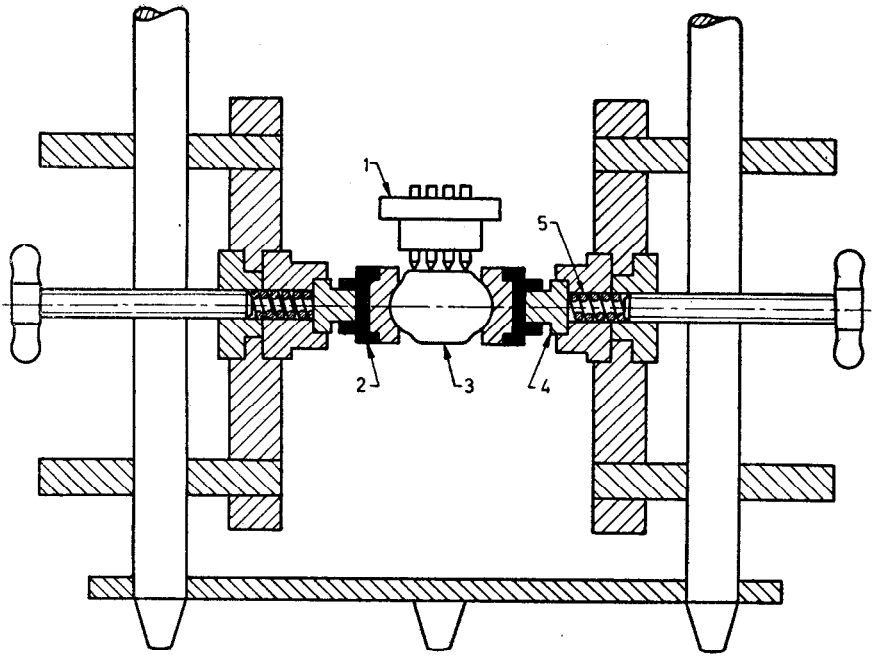
6B Probe Head

FIG. 6 FOUR-PROBE INSTRUMENT — *Contd*

5. PROCEDURE

5.1 For Two-Electrode Method

5.1.1 *Calibration of the Apparatus* — Calibrate the measuring bridge or potentiometer using standard resistance; 0·001 ohm for low and 10 ohms for high resistance. Check the contact resistance of the stainless steel electrodes of the conductivity cell as described below after cleaning the contact (flat) surfaces with alcohol or acetone.



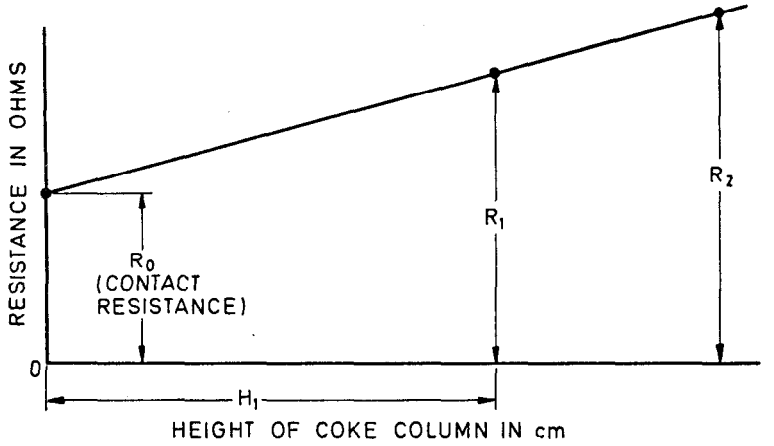
1. Probe head
2. Shaped rubber faced clamping jaws
3. Specimen of carbon or coke
4. Clamping jaw axis
5. Spring

6C Briquette Holder

FIG. 6 FOUR-PROBE INSTRUMENT

5.1.2 *Determination of Contact Resistance and Electrical Resistivity* — Carefully charge a specific quantity of the test sample (*see* **4.1**) into the cell, using a funnel to avoid adhering of particles to the insulating sides of the cell. Place the charged cell on a rotary or syntron vibrator for half a minute to consolidate the charge. Then put the cell on the flat surface of the spindle of a hydraulic jack, place the upper electrode on the coke mass and compress it by applying a pressure of 4·25 MPa (42·5 kgf/cm^2) for 10 to 15 minutes till the dial gauge shows no further change in the height of the coke column in the cell. Note the height (H) in cm of the coke column from the dial gauge. Then pass a definite current through the coke column, from a storage battery or cell and a rheostat and note the resistance. Then reverse the direction of the flow of current and again

note the resistance and take the mean of the two values. Carry out duplicate determinations as above using increasing quantities of the test sample and plot a curve of resistance values for increasing column heights of the test sample. Extrapolate the curve which is a straight line, to meet the resistance axis as shown below and obtain contact resistance:



5.1.2.1 Calculate the electrical resistivity P as follows:

$$P = (R_1 - R_0)\frac{Q}{H_1} \text{ ohm.cm}$$

where

R_1 = resistance in ohms of the coke column of height H_1 cm;

R_0 = contact resistance in ohms, obtained from the graph in **5.1.2**; and

Q = cross-sectional area of the coke column in cm^2.

5.2 For Four-Probe Method — Fix the specimen (*see* **4.2**) into the specimen holder with the ground face upwards to accommodate the four probes. If necessary, rubber faced recessed plates may be employed for holding the specimen firmly in the case of non-uniform shapes. Set the four probes on the flat surface of the specimen firmly to ensure proper contact.

5.2.1 Depending on the resistivity of the specimen, pass a current up to 250 mA through probes 1 and 4 and measure the potential difference between the probes 2 and 3 using the potentiometer or the vacuum tube microvoltmeter and calculate resistivity as prescribed in **2.2**.

6. RESULTS

6.1 The results shall be computed from the arithmetic mean of duplicate determinations, and shall be expressed in terms of ohm.centimetres.

7. REPORT

7.1 The report shall include the following information:

a) Identification and previous history of the sample,

b) Ambient temperature in degrees Celsius at the time of the test, and

c) Method used for determining electrical resistivity.

8. PRECISION OF THE METHOD

8.1 Repeatability — The results of duplicate determinations, carried out at different times in the same laboratory, by the same operator, with the same apparatus, on representative samples drawn from the same lot, shall not differ by more than 2 percent.

8.2 Reproducibility — The means of the results of determinations carried out in different laboratories on representative portions drawn from the same lot, shall not differ by more than 4 percent.

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